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(12) **United States Patent**
Gosavi et al.(10) **Patent No.:** **US 9,422,194 B2**(45) **Date of Patent:** **Aug. 23, 2016**(54) **WIDE TEMPERATURE RANGE CEMENT RETARDER**(71) Applicant: **Halliburton Energy Services, Inc.,**
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Houston, TX (US)(*) Notice: Subject to any disclaimer, the term of this
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U.S.C. 154(b) by 0 days.(21) Appl. No.: **15/061,638**(22) Filed: **Mar. 4, 2016**(65) **Prior Publication Data**

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Related U.S. Application Data(62) Division of application No. 13/458,718, filed on Apr.
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CPC C04B 2103/22; C04B 2103/46;
C04B 40/06; C04B 2103/20; C09K 8/42;
C09K 8/467; C09K 8/487
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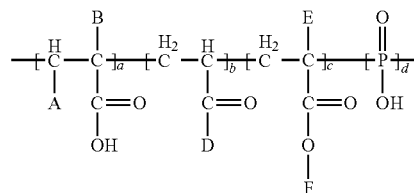
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Primary Examiner — Angela M DiTrani(74) *Attorney, Agent, or Firm* — Craig W. Roddy; Baker
Botts L.L.P.(57) **ABSTRACT**

According to an embodiment, a composition that can be
 used in a cementing. The composition includes at least:

- (i) a hydraulic cement; and
 (ii) a copolymer comprising at least the monomeric units
 (a) through (d) of the following formula:

(a) A = H, B = H, CH₃ (or) A = COOH, B = H, CH₃(b) D = —NH—C(CH₃)₂—CH₂—SO₃M where M = Na, K(c) E = H, CH₃ F = —(CH₂)₂—OH, —CH₂—CH(CH₃)—OH

wherein the monomeric units (a) through (d) can be any
 sequence and any proportion in the copolymer. With
 water, the composition of the hydraulic cement and
 such a copolymer becomes a cement composition.
 According to the method, the cement composition is
 introduced into a well and allowed to set in the well.

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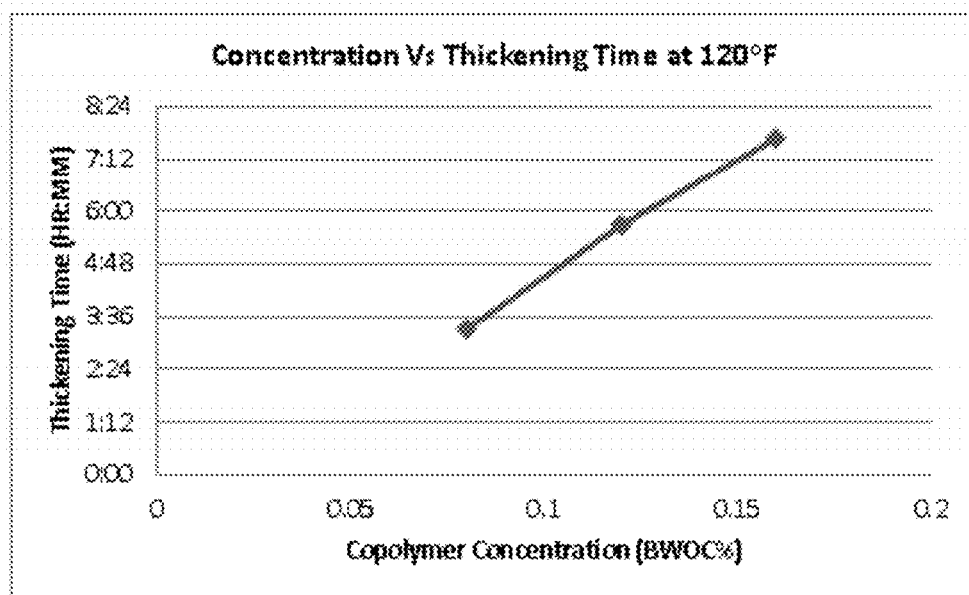


Figure 1. Effect of copolymer (retarder) concentration on thickening time at 120 °F

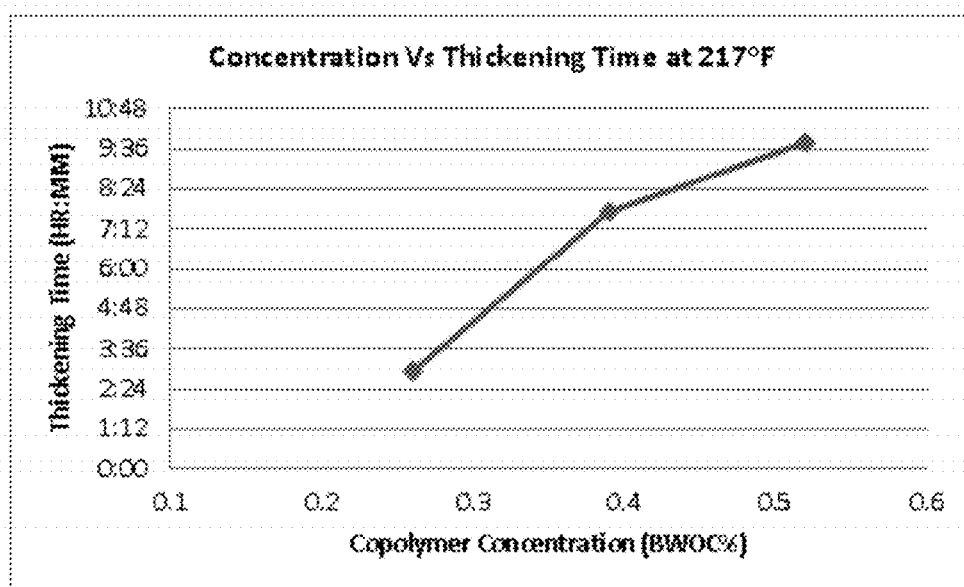


Figure 2. Effect of copolymer (retarder) concentration on thickening time at 217 °F

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WIDE TEMPERATURE RANGE CEMENT RETARDER

CROSS-REFERENCE TO RELATED APPLICATIONS

This is a divisional of U.S. application Ser. No. 13/458, 718 filed on Apr. 27, 2012, the entirety of which is incorporated herein by reference.

TECHNICAL FIELD

The disclosures are in the field of producing crude oil or natural gas from subterranean formations. More specifically, the disclosures generally relate to cementing in oil or gas wells.

BACKGROUND ART

In the context of production from a well, oil and gas are understood to refer to crude oil and natural gas. Oil and gas are naturally occurring hydrocarbons in certain subterranean formations.

A subterranean formation is a body of rock that has sufficiently distinctive characteristics and is sufficiently continuous for geologists to describe, map, and name it. A subterranean formation having a sufficient porosity and permeability to store and transmit fluids is sometimes referred to as a reservoir. A subterranean formation containing oil or gas may be located under land or under the seabed off shore. Oil and gas reservoirs are typically located in the range of a few hundred feet (shallow reservoirs) to a few tens of thousands of feet (ultra-deep reservoirs) below the surface of the land or seabed.

To produce oil or gas, a well is drilled into a subterranean formation that is an oil or gas reservoir. A well includes a wellhead and at least one wellbore from the wellhead penetrating the earth.

Typically, a wellbore must be drilled hundreds or thousands of feet into the earth to reach an oil or gas bearing formation. Generally, the greater the depth of the formation the higher the static temperature and pressure of the formation.

Generally, well services include a wide variety of operations that may be performed in oil, gas, geothermal, or water wells, such as drilling, cementing, completion, and intervention. These well services are designed to facilitate or enhance the production of desirable fluids such as oil or gas from or through a subterranean formation.

In general, drilling is the process of drilling the wellbore. After the hole is drilled, sections of steel pipe, referred to as casing, which are slightly smaller in diameter than the borehole, are placed in at least the uppermost portions of the wellbore. The casing provides structural integrity to the newly drilled borehole.

Cementing is a common well operation. For example, cement compositions can be used in cementing operations in which a string of pipe, such as casing or liner, is cemented in a wellbore. After setting, the cement stabilizes the pipe in the wellbore and prevents undesirable migration of fluids along the annulus between the wellbore and the outside of the casing or liner between various zones of subterranean formations penetrated by the wellbore. Where the wellbore penetrates into a hydrocarbon-bearing zone of a subterranean formation, the casing can later be perforated to allow fluid communication between the zone and the wellbore. The cemented casing also enables subsequent or remedial

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separation or isolation of one or more production zones of the wellbore, for example, by using downhole tools such as packers or plugs, or by using other techniques, such as forming sand plugs or placing cement in the perforations. Cement compositions can also be utilized in intervention operations, such as in plugging highly permeable zones or fractures in zones that may be producing too much water, plugging cracks or holes in pipe strings, and the like.

After drilling and cementing the casing, completion is the process of making a well ready for production or injection. This principally involves preparing a zone of the wellbore to the required specifications, running in the production tubing and associated downhole equipment, as well as perforating and stimulating as required.

Intervention is any operation carried out on a well during or at the end of its productive life that alters the state of the well or well geometry, provides well diagnostics, or manages the production of the well. Workover can broadly refer to any kind of well intervention that involves invasive techniques, such as wireline, coiled tubing, or snubbing. More specifically, though, workover refers to the process of pulling and replacing a completion.

A well service usually involves introducing a well fluid into a well. As used herein, a "well fluid" is a fluid used in a well service. As used herein, a "well fluid" broadly refers to any fluid adapted to be introduced into a well for any purpose. A well fluid can be, for example, a drilling fluid, a cement composition, a treatment fluid, or a spacer fluid. If a well fluid is to be used in a relatively small volume, for example less than about 200 barrels (32 m³), it is sometimes referred to in the art as a wash, dump, slug, or pill.

Hydraulic cement is a material that when mixed with water hardens or sets over time because of a chemical reaction with the water. The cement composition sets by a hydration process, and it passes through a gel phase to solid phase. Because this is a chemical reaction with the water, hydraulic cement is capable of setting even under water. The hydraulic cement, water, and any other components are mixed to form a cement composition in the initial state of slurry, which should be a fluid for a sufficient time before setting for pumping the composition into the wellbore and for placement in a desired downhole location in the well.

In performing cementing, a cement composition is pumped as a fluid (typically in the form of suspension or slurry) into a desired location in the wellbore. For example, in cementing a casing or liner, the cement composition is pumped into the annular space between the exterior surfaces of a pipe string and the borehole (that is, the wall of the wellbore). The cement composition is allowed time to set in the annular space, thereby forming an annular sheath of hardened, substantially impermeable cement. The hardened cement supports and positions the pipe string in the wellbore and fills the annular space between the exterior surfaces of the pipe string and the borehole of the wellbore.

It is important to maintain a cement in a pumpable slurry state until it placed in a desired portion of the well. For this purpose, a cement retarder, which is sometimes referred to as a set retarder or simply a retarder, can be used in a cement composition. A retarder retards the setting process and helps provide adequate pumping time to place the cement slurry.

Without being limited by any theory, it is believed a retarder works by one or more of the principles of chelation, adsorption, or precipitation.

In general, the selection of a cement retarder depends upon the well temperature. In addition, different thickening time can be achieved at particular temperature by varying the concentration of the retarder in the cement composition.

be understood to set forth every number and range encompassed within the broader range of values.

Terms such as “first,” “second,” “third,” etc. are assigned arbitrarily and are merely intended to differentiate between two or more components, parts, or steps that are otherwise similar or corresponding in nature, structure, function, or action. For example, the words “first” and “second” serve no other purpose and are not part of the name or description of the following name or descriptive terms. The mere use of the term “first” does not require that there be any “second” similar or corresponding component, part, or step. Similarly, the mere use of the word “second” does not require that there be any “first” or “third” similar or corresponding component, part, or step. Further, it is to be understood that the mere use of the term “first” does not require that the element or step be the very first in any sequence, but merely that it is at least one of the elements or steps. Similarly, the mere use of the terms “first” and “second” does not necessarily require any sequence. Accordingly, the mere use of such terms does not exclude intervening elements or steps between the “first” and “second” elements or steps, etc.

Well Terms

A “subterranean formation” is a body of rock that has sufficiently distinctive characteristics and is sufficiently continuous for geologists to describe, map, and name it.

A subterranean formation having a sufficient porosity and permeability to store and transmit fluids is sometimes referred to as a “reservoir.”

A “well” includes a wellhead and at least one wellbore from the wellhead penetrating the earth. The “wellhead” is the surface termination of a wellbore, which surface may be on land or on a seabed. A “well site” or “job site” is the geographical location of a wellhead of a well. It may include related facilities, such as a tank battery, separators, compressor stations, heating or other equipment, and fluid pits. If offshore, a well site can include a platform.

The “wellbore” refers to the drilled hole, including any cased or uncased portions of the well. The “borehole” usually refers to the inside wellbore wall, that is, the rock face or wall that bounds the drilled hole. A wellbore can have portions that are vertical, horizontal, or anything in between, and it can have portions that are straight, curved, or branched. As used herein, “uphole,” “downhole,” and similar terms are relative to the direction of the wellhead, regardless of whether a wellbore portion is vertical or horizontal.

As used herein, introducing “into a well” means introduced at least into and through the wellhead. According to various techniques known in the art, tubulars, equipment, tools, or well fluids can be directed from the wellhead into any desired portion of the wellbore. Additionally, a well fluid can be directed from a portion of the wellbore into the rock matrix of a zone.

As used herein, the word “tubular” means any kind of pipe. Examples of tubulars include, but are not limited to, a drill pipe, a casing, a tubing string, a line pipe, and a transportation pipe. Tubulars can also be used to transport fluids into or out of a subterranean formation, such as oil, gas, water, liquefied methane, coolants, and heated fluids. For example, a tubular can be placed underground to transport produced hydrocarbons or water from a subterranean formation to another location.

As used herein, the term “annulus” means the space between two generally cylindrical objects, one inside the other, where fluid can flow. The objects can be concentric or eccentric. One of the objects can be a tubular and the other object can be an enclosed conduit. The enclosed conduit can

be a wellbore or borehole or it can be another tubular. The following examples illustrate some situations in which an annulus can exist, but are in no way limiting as to all the situations in which an annulus can exist. Referring to an oil, gas, or water well, in an open hole well, the space between the wellbore and the outside of a tubing string is an annulus. In a cased hole, the space between the wellbore and the outside of the casing is an annulus. Also, in a cased hole, there may be an annulus between the tubing string and the inside of the casing.

As used herein, a “well fluid” broadly refers to any fluid adapted to be introduced into a well for any purpose. A well fluid can be, for example, a drilling fluid, a cement composition, a treatment fluid, or a spacer fluid. If a well fluid is to be used in a relatively small volume, for example less than about 200 barrels (about 32 m³), it is sometimes referred to in the art as a wash, dump, slug, or pill.

Broadly, a zone refers to an interval of rock along a wellbore that is differentiated from uphole and downhole zones based on hydrocarbon content or other features, such as permeability, composition, perforations or other fluid communication with the wellbore, faults, or fractures. A zone of a wellbore that penetrates a hydrocarbon-bearing zone that is capable of producing hydrocarbon is referred to as a “production zone.” As used herein, a “treatment zone” refers to an interval of rock along a wellbore into which a well fluid is directed to flow from the wellbore.

The term “design temperature” refers to an estimate or measurement of the actual temperature at the down hole environment at the time of a well treatment. That is, design temperature takes into account not only the bottom hole static temperature (“BHST”), but also the effect of the temperature of the well fluid on the BHST during treatment, which is the bottom hole circulation temperature (“BHCT”). Because treatment fluids may be considerably cooler than BHST, the difference between the two temperatures can be quite large. Ultimately, if left undisturbed, a subterranean formation will return to the BHST.

Fluid Terms

The physical state or phase of a substance (or mixture of substances) and other physical properties are determined at a temperature of 77° F. (25° C.) and a pressure of 1 atmosphere (Standard Laboratory Conditions) without any applied shear.

As used herein, a fluid is a substance that behaves as a fluid under Standard Laboratory Conditions.

Every fluid inherently has at least a continuous phase. A fluid can have more than one phase. The continuous phase of a well fluid is a liquid under standard laboratory conditions. For example, a well fluid can in the form of be a suspension (solid particles dispersed in a liquid phase), an emulsion (liquid particles dispersed in another liquid phase), or a foam (a gas phase dispersed in liquid phase).

Unless otherwise specified, the apparent viscosity of a fluid (excluding any suspended solid particulate larger than silt) is measured with a Fann Model 50 type viscometer using an R1 rotor, B1 bob, and F1 torsion spring at a shear rate of 40 1/s, and at a temperature of 77° F. (25° C.) and a pressure of 1 atmosphere. For reference, the viscosity of pure water is about 1 cP.

Cement Compositions

As used herein, a “cement composition” is a mixture of at least hydraulic cement and water. The cement composition can also include additives.

As used herein, the term “cement” means a dry particulate (e.g., powder) substance that acts as a binder to bind other materials together. A “hydraulic cement” (e.g., Portland

percent by weight of cement, alternatively from about 36 to about 60 percent by weight of cement.

It is recognized that, in general, for water to be suitable for use in a well fluid, usually all that is required is that the water does not contain one or more materials that would be particularly detrimental to the chemistry of the cement composition or detrimental to downhole equipment or the subterranean formation.

The aqueous phase can include freshwater or non-freshwater. Non-freshwater sources of water can include surface water ranging from brackish water to seawater, brine, returned water (sometimes referred to as flowback water) from the delivery of a well fluid into a well, unused well fluid, and produced water. As used herein, brine refers to water having at least 40,000 mg/L total dissolved solids.

Additives

Cement composition can contain additives. Such additives may include but not limited to resins, latex, stabilizers, silica, microspheres, aqueous superabsorbers, viscosifying agents, suspending agents, dispersing agents, salts, accelerants, surfactants, retardants, defoamers, high-density materials, low-density materials, fluid loss control agents, elastomers, vitrified shale, gas migration control additives, formation conditioning agents, or other additives or modifying agents, and/or combinations thereof.

Method Steps

A cement composition can be prepared at the well site, prepared at a plant or facility prior to use, or certain components can be pre-mixed prior to use and then transported to the well site. Certain components of the treatment fluid may be provided as a "dry mix" to be combined with other components prior to or during introducing into the well.

In certain embodiments, the preparation of a cement composition according to the present disclosure can be done at the well site in a method characterized as being performed "on the fly." The term "on-the-fly" includes methods of combining two or more components wherein a flowing stream of one element is continuously introduced into flowing stream of another component so that the streams are combined and mixed while continuing to flow as a single stream as part of the on-going treatment. Such mixing can also be described as "real-time" mixing.

As used herein, introducing "into a well" means introduced at least into and through the wellhead. According to various techniques known in the art, equipment, tools, or well fluids can be directed from the wellhead into any desired portion of the wellbore. Additionally, a well fluid can be directed from a portion of the wellbore into the rock matrix of a zone.

As used herein, "into a treatment zone" means into and through the wellhead and, additionally, through the wellbore and into the treatment zone.

Often the step of delivering a well fluid into a well is within a relatively short period after forming the well fluid, e.g., less within 30 minutes to one hour. More preferably, the step of delivering the well fluid is immediately after the step of forming the well fluid, which is "on the fly."

It should be understood that the step of delivering a well fluid into a well can advantageously include the use of one or more fluid pumps.

In an embodiment, after the step of introducing, the method includes the step of allowing time for the cement composition to set in the well.

Preferably, after the step of allowing time for setting, the method includes a step of producing oil or gas from the well.

Pumping Time

As used herein, the "pumping time" is the total time required for pumping a hydraulic cement composition into a desired portion or zone of the well, plus a safety factor, in a cementing operation.

Thickening Time

As used herein, the "thickening time" is how long it takes for a cement composition to become unpumpable at a specified temperature and specified pressure. The pumpability of a cement composition is related to the consistency of the composition. The consistency of a cement composition is measured in Bearden units of consistency (Bc), a dimensionless unit with no direct conversion factor to the more common units of viscosity. As used herein, a cement composition becomes "unpumpable" when the consistency of the composition reaches 70 Bc.

As used herein, the consistency of a cement composition is measured according to ANSI/API Recommended Practice 10B-2 as follows. The cement composition is mixed. The cement composition is then placed in the test cell of a High-Temperature, High-Pressure (HTHP) consistometer, such as a Fann Model 275 or a Chandler Model 8240. The cement composition is tested in the HTHP consistometer at the specified temperature and pressure. Consistency measurements are taken continuously until the consistency of the cement composition exceeds 70 Bc.

Of course, the thickening time should be greater than the designed pumping time for a cementing operation.

Setting and Compressive Strength

As used herein, the term "set" is intended to mean the process of becoming hard or solid by curing. Depending on the cement composition and conditions, it can take a few minutes to 72 hours or longer for some cement compositions to initially set. A cement composition sample that is at least initially set is suitable for destructive compressive strength testing and permeability testing. Some cement compositions can continue to develop a compressive strength greater than 50 psi over the course of several days. The compressive strength of certain kinds of cement compositions can reach over 10,000 psi.

The compressive strength of a cement composition can be used to indicate whether the cement composition has set. As used herein, a cement composition is considered "initially set" when the cement composition has developed a compressive strength of 50 psi using the non-destructive compressive strength method. As used herein, the "initial setting time" is the time between when the cement is added to the water and when the cement composition is initially set. If not otherwise stated, the setting and the initial setting time is determined at a temperature of 212° F. and a pressure of 3,000 psi.

Compressive strength is generally measured at a specified time after the cement composition has been mixed and then cured at a specified temperature and pressure. For example, compressive strength can be measured at a time in the range of about 24 to about 48 hours at a temperature of 212° F. According to ANSI/API Recommended Practice 10B-2, compressive strength can be measured by either a destructive method or non-destructive method.

The destructive method mechanically tests the strength of cement composition samples at various points in time by crushing the samples in a compression-testing machine. The destructive method is performed as follows. The cement composition is mixed. Then, the mixed composition is cured. The cured cement composition sample is placed in a compressive strength testing device, such as a Super L Universal testing machine model 602, available from Tinius Olsen, Horsham in Pennsylvania, USA. According to the destructive method, the compressive strength is calculated as the force required to break the sample divided by the

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(b) D = $\text{—NH—C(CH}_3)_2\text{—CH}_2\text{—SO}_3\text{M}$ where M = Na, K

(c) E = H, CH₃ F = —(CH₂)₂—OH, —CH₂—CH(CH₃)—OH

2. The composition according to claim 1, wherein the hydraulic cement is a cementitious material selected from the group consisting of Portland cements, pozzolanic cements, gypsum cements, phosphate cements, high alumina content cements, silica cements, high alkalinity cements, shale cements, acid/base cements, magnesia cements, fly ash cement, zeolite cement systems, cement kiln dust cement systems, slag cements, micro-fine cement, metakaolin, or combinations thereof.

4. The composition according to claim 1, wherein the monomeric units (a) through (d) in the copolymer are in the following ranges:

(a) is in the range of 65 to 75% by weight;
(b) is in the range of 10 to 15% by weight;
(c) is in the range of 10 to 15% by weight; and
(d) is in the range of 3 to 7% by weight.

5. The composition according to claim 1, wherein the concentration of the copolymer is between 0.05% by weight of the cement and 3% by weight of the cement.

6. The composition according to claim 1, further comprising silica flour.

7. The composition according to claim 1, further comprising a fluid-loss control additive.

8. The composition according to claim 7, wherein the fluid-loss control additive is selected from the group consisting of: sulfonic acid copolymer, acrylamide copolymers, cellulosic polymers and derivatives thereof, and modified cellulosic polymers.

9. The composition according to claim 1, further comprising a retarder intensifier.

10. The composition according to claim 9, wherein the retarder intensifier is tartaric acid.

11. The composition according to claim 1, further comprising water.

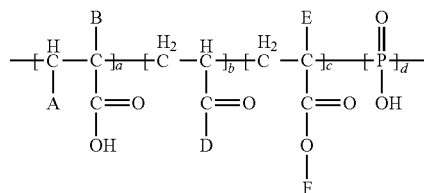
12. The composition according to claim **11**, wherein the composition has a thickening time of at least 2 hours at any temperature between 100° F. and 360° F.

13. A composition comprising:

(i) a hydraulic cement; and

(ii) a copolymer comprising at least the monomeric units (a) through (d) of the following formula:

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(a) A = H, B = H, CH₃ (or) A = COOH, B = H, CH₃

(b) D = —NH—C(CH₃)₂—CH₂—SO₃M where M = Na, K

(c) E = H, CH₃ F = —(CH₂)₂—OH, —CH₂—CH(CH₃)—OH

wherein the monomeric units (a) through (d) can be in any sequence in the copolymer; and wherein the monomeric units (a) through (d) in the copolymer are in the following ranges:

(a) is in the range of 65 to 75% by weight;

(b) is in the range of 10 to 15% by weight;

(c) is in the range of 10 to 15% by weight; and

(d) is in the range of 3 to 7% by weight.

14. The composition according to claim 13, wherein the hydraulic cement is a cementitious material selected from the group consisting of Portland cements, pozzolanic

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cements, gypsum cements, phosphate cements, high alumina content cements, silica cements, high alkalinity cements, shale cements, acid/base cements, magnesia cements, fly ash cement, zeolite cement systems, cement kiln dust cement systems, slag cements, micro-fine cement, metakaolin, or combinations thereof.

15. The composition according to claim 13, wherein the monomeric units (a) through (d) are acrylic acid, 2-acrylamido-2-methylpropane sulfonic acid sodium salt, 2-hydroxypropyl acrylate, and phosphinic acid, respectively.

16. The composition according to claim 13, further comprising silica flour.

17. The composition according to claim 13, further comprising a fluid-loss control additive.

18. The composition according to claim 17, wherein the fluid-loss control additive is selected from the group consisting of: sulfonic acid copolymer, acrylamide copolymers, cellulosic polymers and derivatives thereof, and modified cellulosic polymers.

19. The composition according to claim 13, further comprising a retarder intensifier.

20. The composition according to claim 13, further comprising water.

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